THE POWDER METALLURGY OF URANIUM

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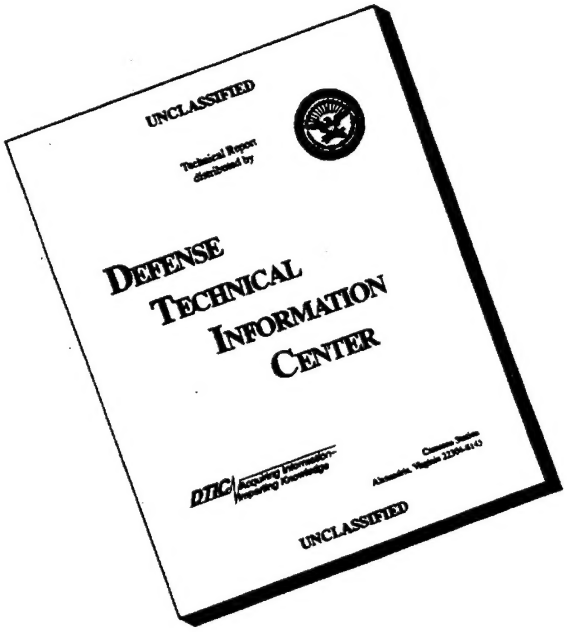

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THE POWDER METALLURGY OF URANIUM

by

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Uranium is a ductile metal and cannot be comminuted to a fine powder by any mechanical means such as crushing, milling or grinding. Uranium, however, reacts readily with hydrogen and forms UH_3, which is a fine powder of less than 400 mesh screen size. The factors controlling the rate of the hydride formation are:

a) the surface area of the metal

b) the temperature at which the reaction takes place

c) the pressure of hydrogen

In order to increase the reaction area, one has to hydride small metal pieces rather than a single mass. The hydrogen reacts with uranium metal at temperatures as low as 100° to 150°C, and the reaction rate becomes quite rapid at approximately 225°C. The hydrogen for this purpose has to be of high purity and any small amount of oxygen in hydrogen delays the start of the reaction. Fig. 1 shows the rate of reaction with the metal in wire form, as a function of the temperature, and further shows that the maximum reaction rate is around 225°C. The hydrogen pressure also affects the rate of reaction; higher pressures result in higher reaction rates. The reaction is exothermic, but since temperatures above 225°C cause a

decrease in rate, the reaction is self-controlling. The fine hydride powder is then decomposed *in vacuo* at temperatures of 225°C or above. The decomposition rate is a function of the temperature, and Fig. 2 shows how the dissociation pressure of UH_3 increases with the decomposition temperature. During decomposition at higher temperatures, the uranium powder particles formed during the decomposition may sinter on account of the high surface activity of uranium, and form larger agglomerates, or sintercakes, which are extremely brittle and can be easily mechanically comminuted to the desired powder particle size. Practically any uranium particle is actually an agglomerate of finer ones, as suggested in the electron-microphotograph of 10,000 X magnification (Fig. 3).

Uranium powder is highly pyrophoric and will immediately start to burn when exposed to air or other oxidizing conditions. Besides its pyrophoricity, uranium powder is highly toxic. The powder, therefore, has to be handled in "dry boxes", as shown in Fig. 4, filled with an inert or protective atmosphere, such as argon or helium.

On account of their large ratio of surface area to volume and, therefore, increased friction area between powder particles, uranium powders do not flow readily, and their apparent density is as low as 6 to 8 g/cc, depending on the decomposition conditions and the actual size of the particles. The compressibility of uranium powder, that is, its ability to form compacts under pressure, is, therefore, very low. When compacted at 30 tsi, the "green" density of the compact is as low as 10 to 11.5 g/cc, depending on the particle size and on the shape and dimensions of the compact.

In order to obtain higher densities of uranium powder compacts, the powder is to be compacted in a high-strength steel die in protective atmosphere at fairly high pressures. Fig. 5 shows the effect of compacting pressures up to 100 tsi on the compact density; even at a pressure of 100 tsi, the powder compacts to only 15.5 g/cc, that is, to approximately 80% of the theoretical density of uranium. These compacts contain a large amount of interconnected pores and are characterized, therefore, by a large amount of surface area; when exposed to air, these porous compacts start burning and the pyrophoricity of the "green" compact, although less than that of the uncompacted powder, makes it necessary that the compacts be handled in a protective atmosphere.

Sintering of uranium powder compacts can best be done in vacuo. Fig. 5 indicates the effect of compacting pressure on the density of uranium powder compacts sintered in vacuo at 1095° and 1120°C, respectively. Uranium powder does not compact and sinter as well as other powders. This is shown in Fig. 6, which indicates the effect of sintering temperature on the density of several metal powders, all compacted at 50 tsi. Compared to the other metal powders, all compacted under similar conditions, uranium powder does not densify before reaching a temperature of approximately 85% of the melting temperature, whereas nickel and other powders start to densify at sintering temperatures in the range of 50% of their melting temperatures. From Figs. 5 and 6, it can be seen that even when sintered close to the melting temperature, 1128°C, uranium powders never sinter to the theoretical density of the material.

During sintering, the uranium powder undergoes two phase transformations: at 663°C, from the orthorhombic alpha lattice to the tetragonal beta lattice, and at 764°C, to the body-centered-cubic gamma lattice. Whether these phase transformations hinder or delay densification during sintering is not yet known — they definitely affect the grain size. Uranium compacts sintered at the elevated temperatures where densification occurs are characterized by a coarse grain structure, the grains sometime being as large as 1500 microns in diameter, as shown in the photomicrograph, Fig. 7.

In powder metallurgy, re-pressing is frequently applied in order to get higher densities of the sintered materials. Re-pressing the sintered uranium compacts at high pressures results only in a slight increase in density, as shown in Fig. 8. Annealing the re-pressed and, therefore, cold-worked compacts in the alpha temperature range of uranium, that is, below 663°C, has no effect on the density but severely affects the grain structure. The structure of a re-pressed sample is shown in Fig. 9. During annealing, a fine grain structure develops, as shown in Fig. 10, and grains of approximately 25 to 30 microns in diameter can be obtained. Microscopic examination of the structure, however, shows that these fine grains are merely sub-grains of the former coarse grains and that their orientation is quite similar to that of the coarse grains from which they were formed. X-ray determination reveals that these fine grains deviate in orientation not more than 10 to 15 degrees from the orientation of the former coarse grains.

Uranium powder, however, can be compacted to practically full density when pressed at elevated temperatures. The well-known powder metallurgical

technique of hot pressing is applicable to uranium powders when hot pressing is carried out in a protective atmosphere or in vacuo. Hot pressing or hot compacting has to be done at temperatures close to the alpha-to-beta phase transformation temperature. Fig. 11 shows the effect of compacting temperature from 300° to 700° on the density of uranium powders compacted at 20 tsi. Compacting just below the alpha-to-beta phase transformation temperature results in a complete densification. The difference between sintering below and above 663°C is shown in the grain structure of the hot-pressed compact; compacting in the alpha range results in a fine grain structure consisting of grains in the range of 10 to 30 microns in diameter, whereas compacting in the beta range results in a coarse grain structure, the particle size of the grains depending on the duration of the hot compacting process. The grain size of uranium compacts pressed in the alpha temperature range is shown in Fig. 12.

The powder metallurgy of uranium also offers other interesting aspects. Similar to a powder metallurgical process for zirconium, which shows that it is possible to compact ZrH_2 powders and to combine the decomposition of the hydride with the sintering process, uranium hydride can also be compacted and decomposed under sintering conditions. The high activity of the fine UH_3 particles during the time of decomposition, perhaps also the presence of atomic hydrogen, increases the bonding activity between powder particles and the rate of densification during sintering. Compacted UH_3 powders, however, are extremely brittle and hard to handle, which makes this process rather difficult.

Powder metallurgical prepared uranium is high in hardness and tensile strength and low in ductility. Special heat treatments of the sintered product are necessary to increase ductility and lower hardness. Sintered uranium densified to a high degree is no longer pyrophoric and can be handled in air without burning. For any machining operation, however, precautions must be taken inasmuch as the lathe turnings easily catch fire, and also on account of the toxicity of fine particles.

Attachments - 12 figures

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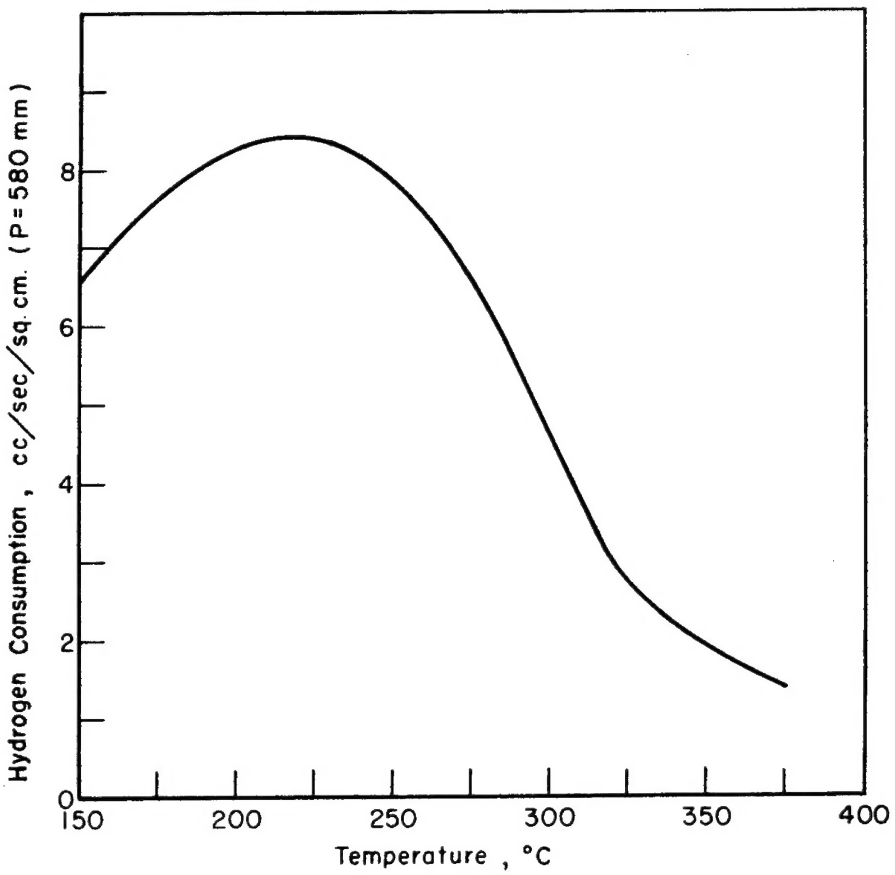


Fig. 1 - The Change in Rate of Reaction of Hydrogen With Uranium as Temperature Increases. Rates are for purified hydrogen reacting with uranium wire.

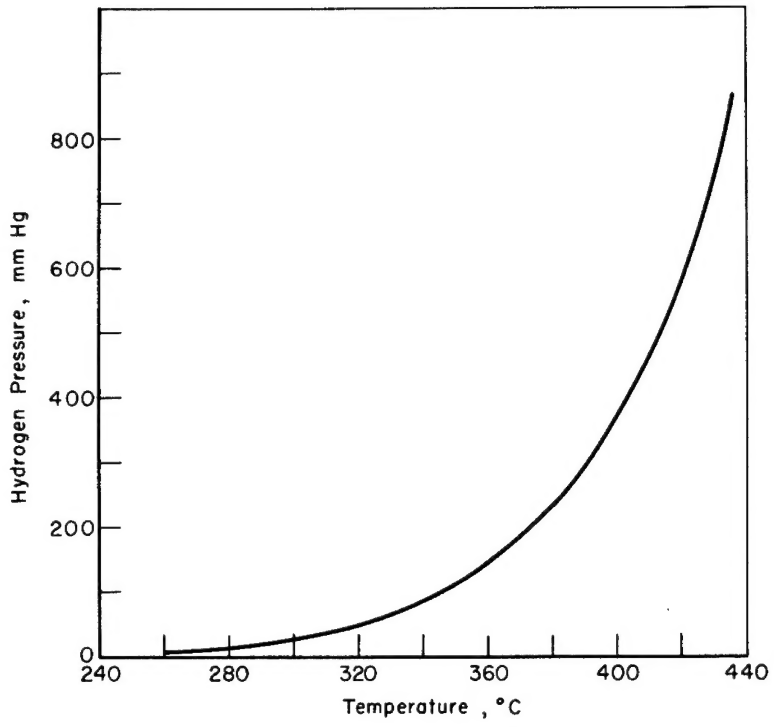


Fig. 2 - Dissociation Pressure of Uranium Hydride Increases With Increasing Temperature.

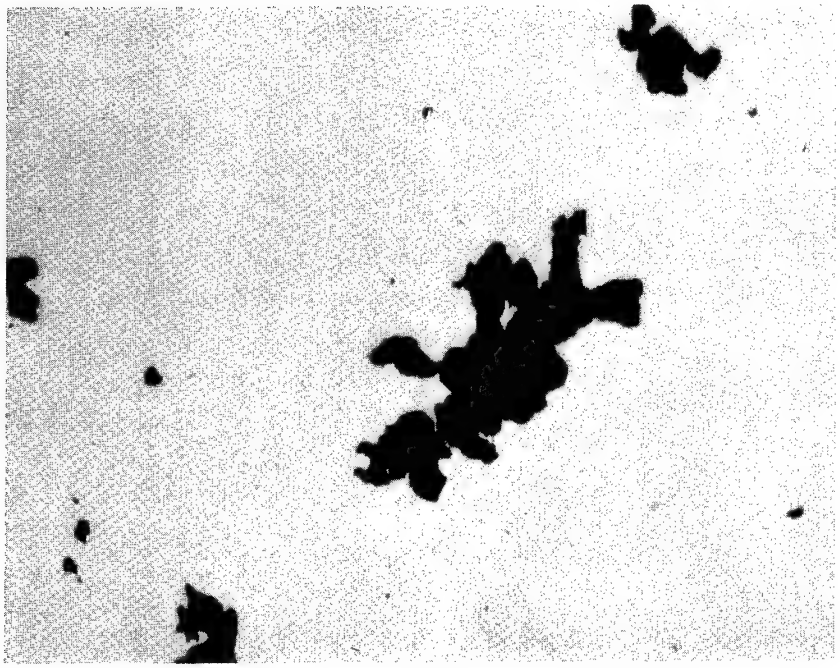
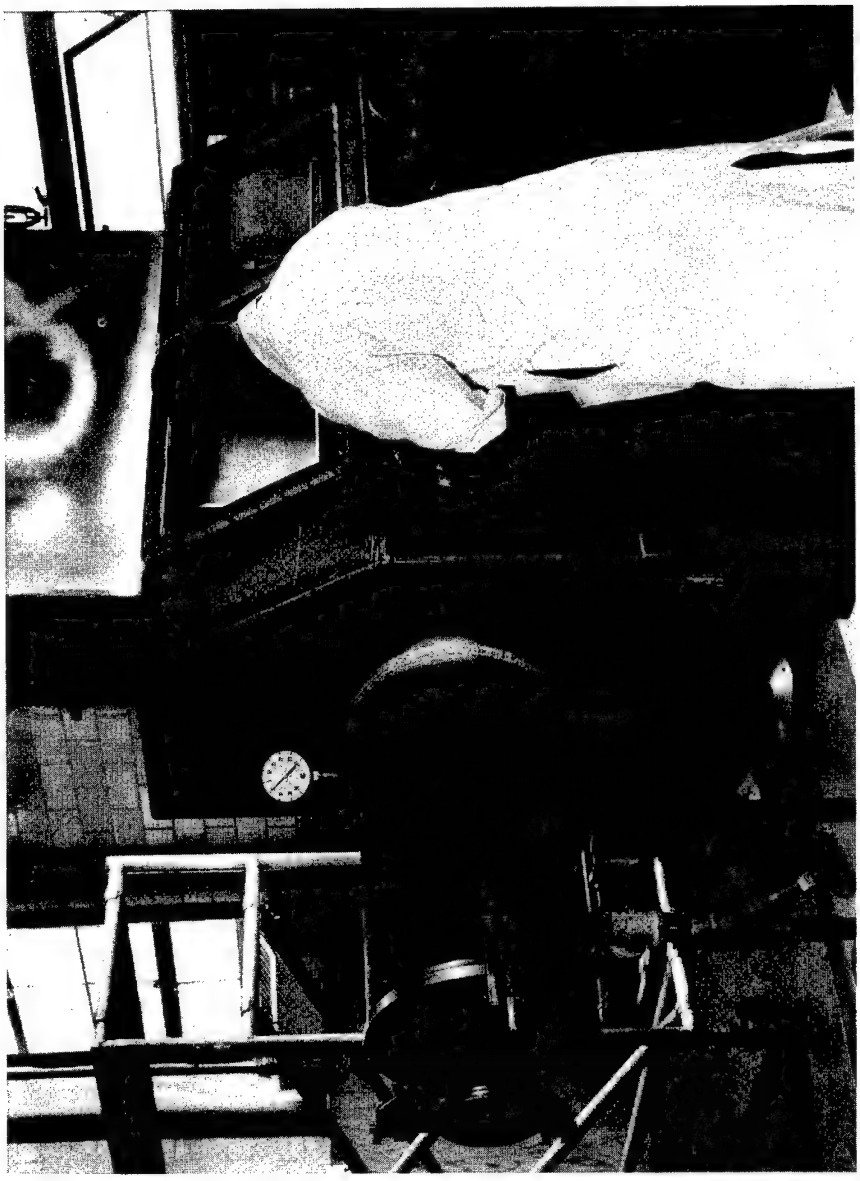


Fig. 3

Plate #1522a X 20,000

Uranium Powder made from UH_3



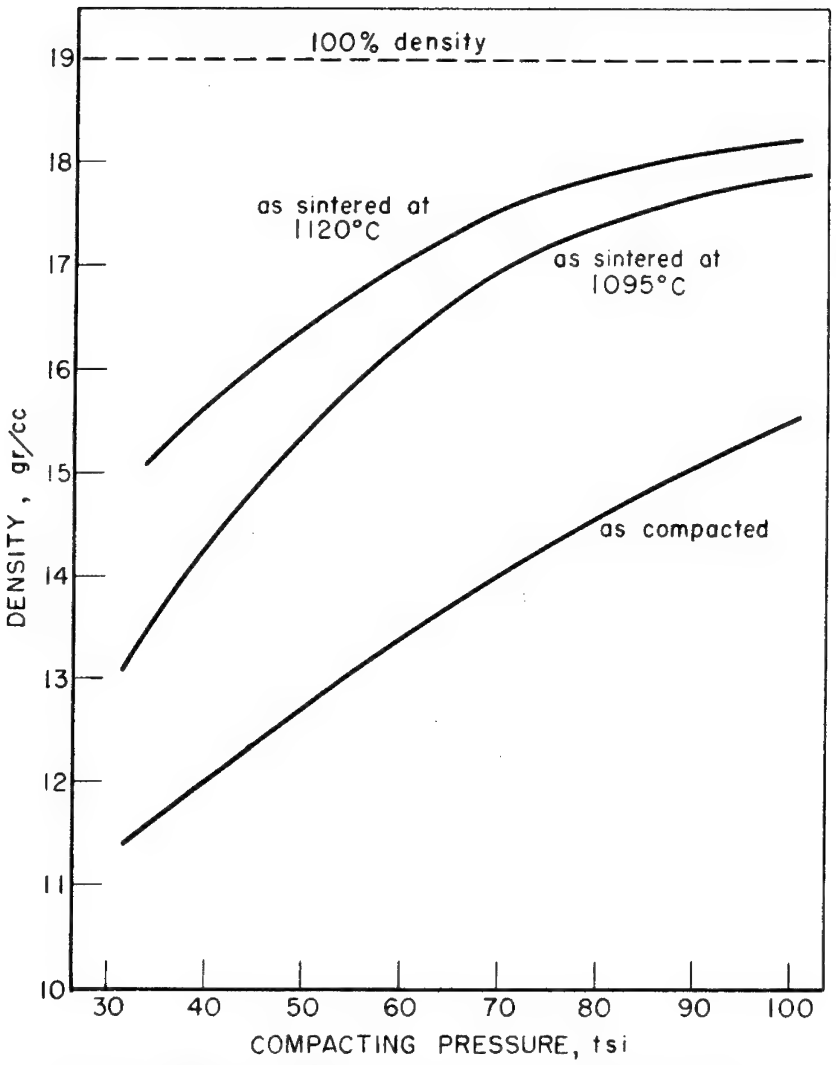


Fig. 5- Effect of compacting pressure on density of "green" and sintered uranium powder compacts.

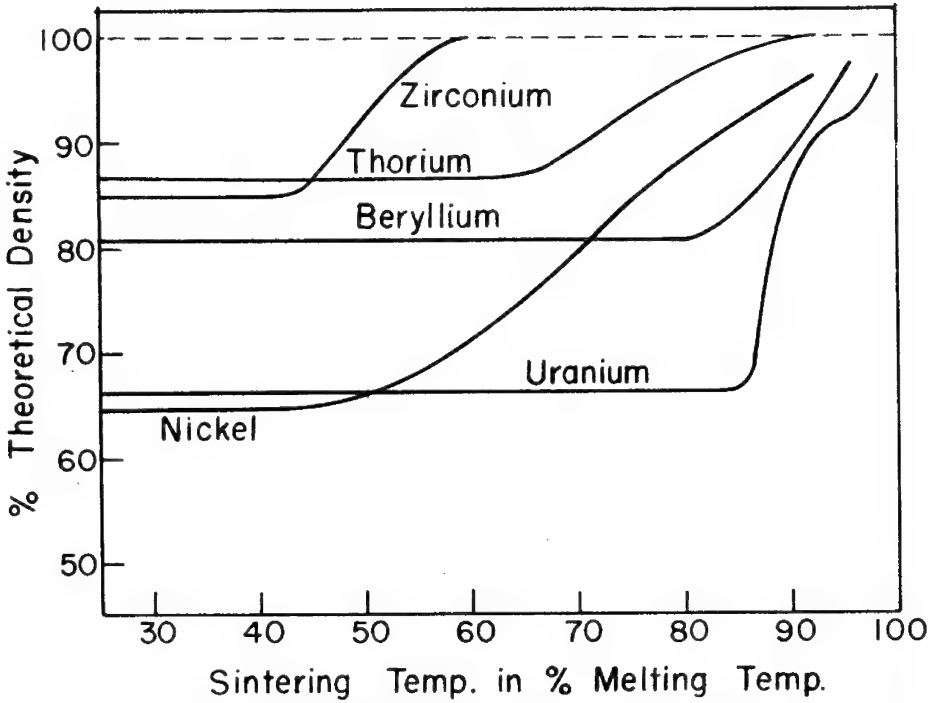


Fig. 6- Effect of sintering temperature on the density of various reactor metals.

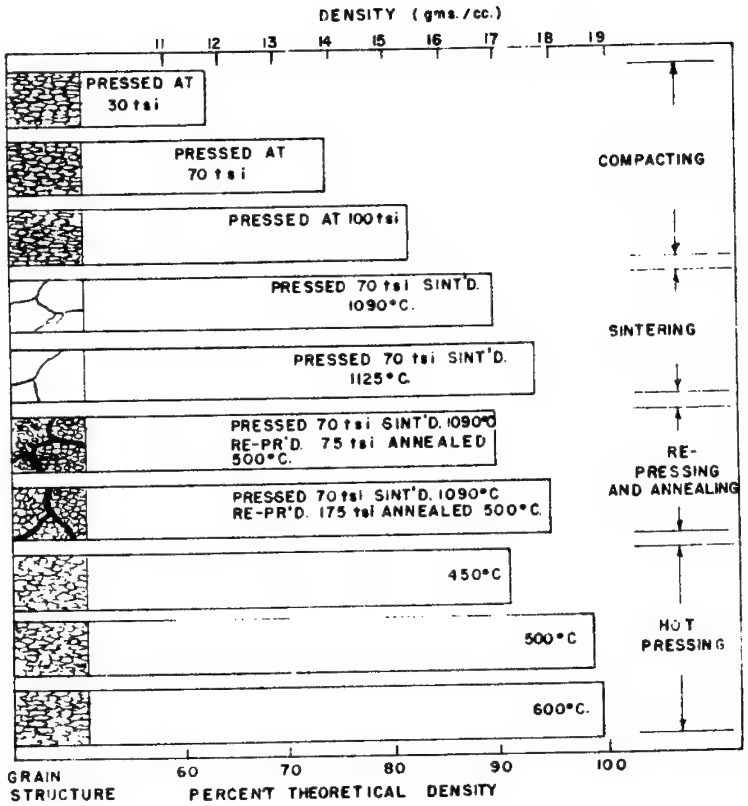
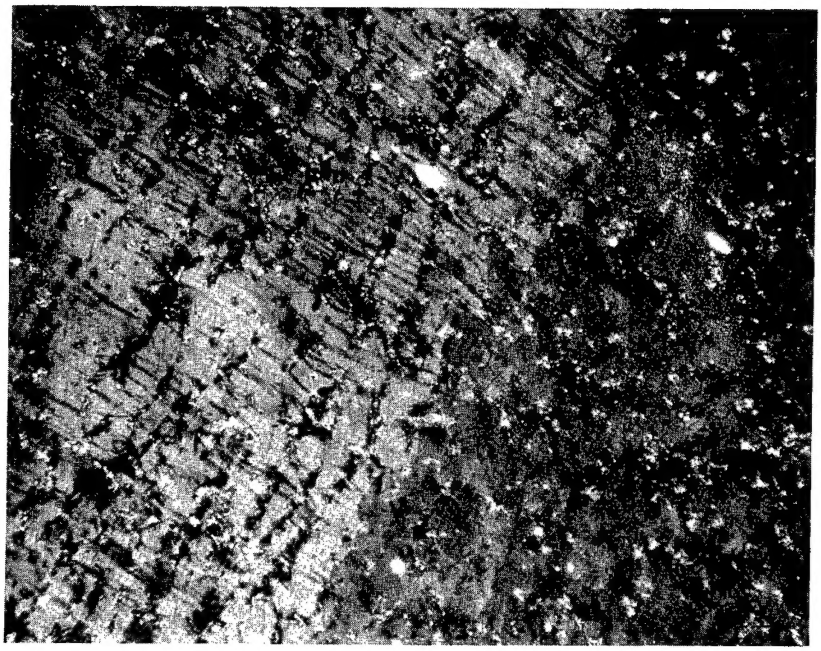
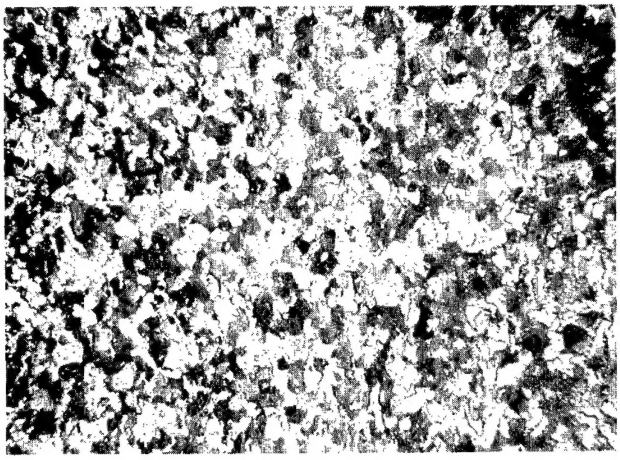


FIG. 1 DENSITY AND GRAIN SIZE OF URANIUM PRODUCED BY POWDER METALLURGY METHODS.





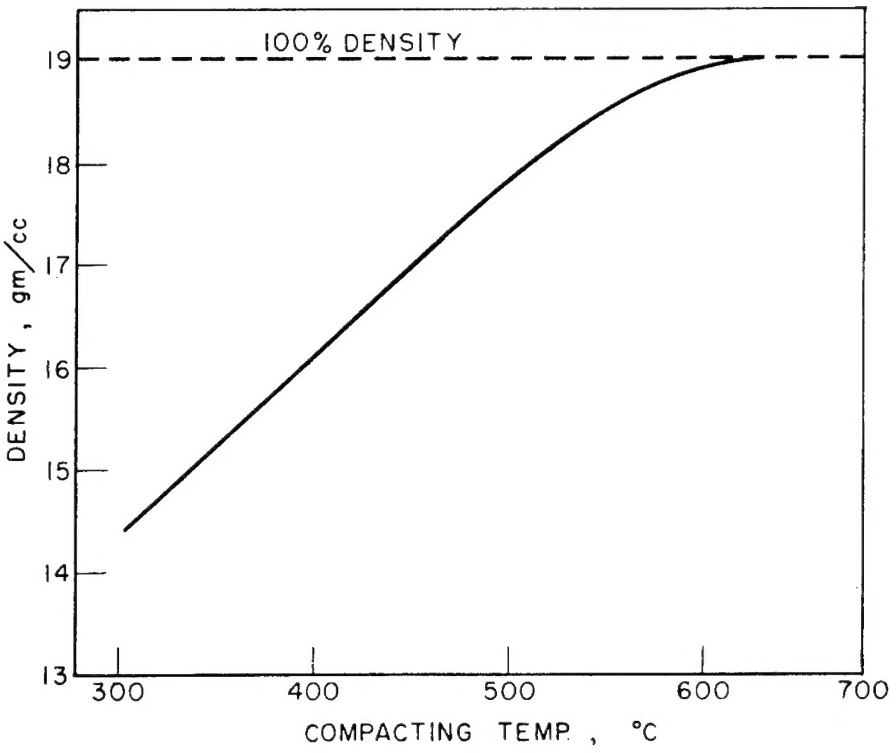


Fig. 11 Density of hot compacted uranium powder vs. compacting temperature (compacting pressure 20 tsi).